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G1S SGX SIA

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US 5463894 A US 5035583 A
WPI Accession No.93-161304/199320 & FR 2681424 A1 (CHOISNET et al.) 19.03.93 (see abstract) WPI Accession No.88-339077/198848 & DE 3741589 C (LOTHMANN) 01.12.88 (see abstract)

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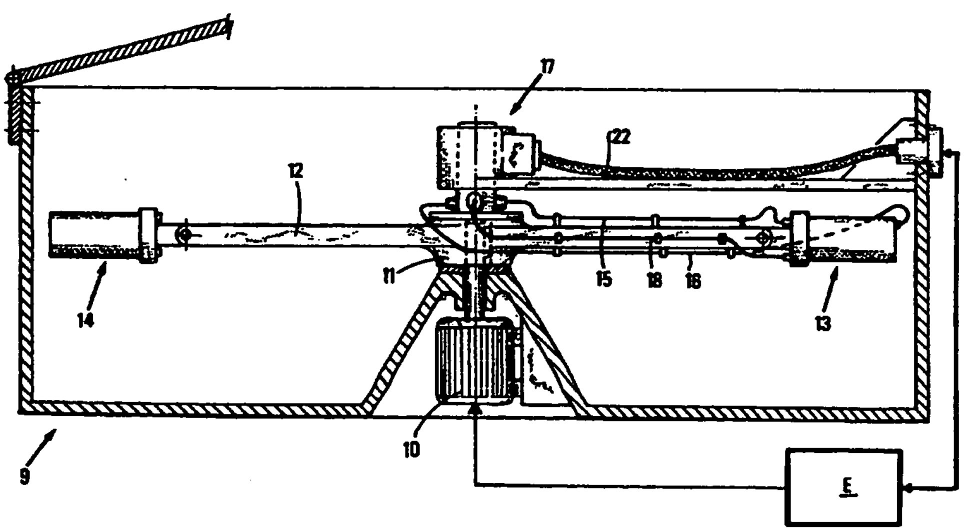
(54) Abstract Title
Measuring physical characteristics of a porous sample

(57) The invention relates to a device for taking measurements of physical characteristics on a porous sample by operating draining and imbibition phases in succession in the presence of a first, electrically conductive fluid (brine) and a second fluid (oil) of a density lower than that of the first fluid by means of a centrifuge, the speed of which successively increases and decreases. The samples saturated with the first fluid is placed in a container 13,14 fastened to the end of an arm 12 driven in rotation by a motor 10 and connected by means of a rotating electro-hydraulic connector 17 to a stationary unit E for controlling and acquiring the measurement signals incorporating hydraulic means for displacing the fluids and an acquisition device connected to a capacitive sensor in the container, which issues signals indicating the position of the interface between the two fluids.

FIG.4



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At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

FIG.1

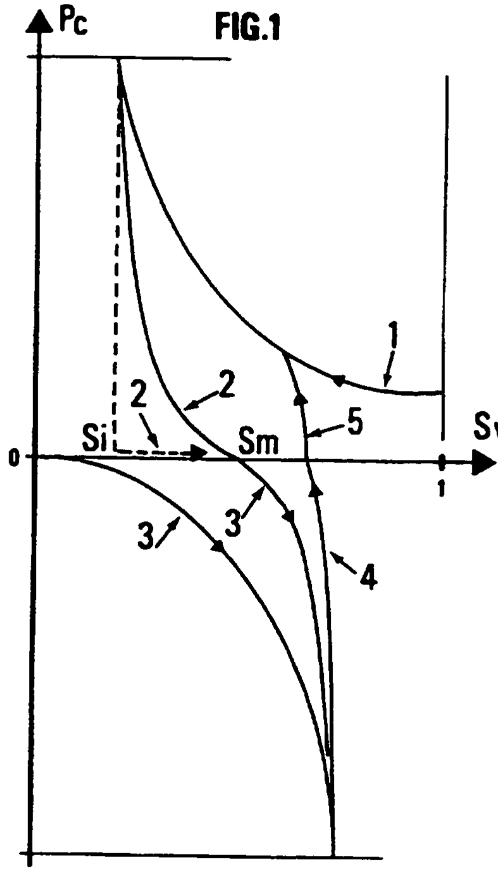


FIG.2

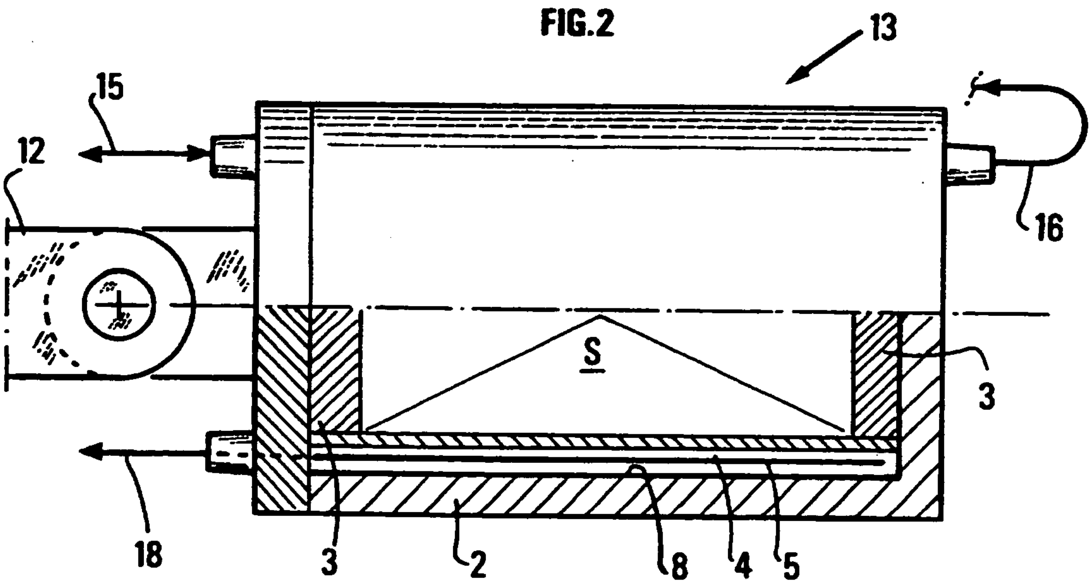


FIG.3

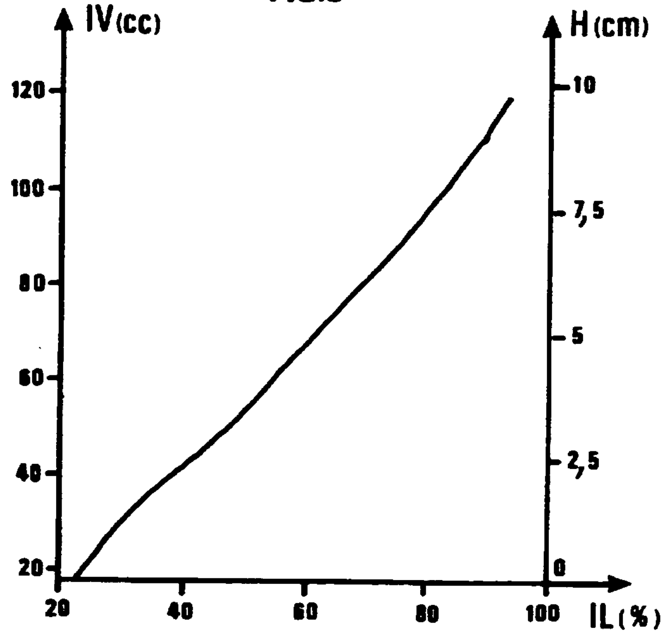


FIG.6

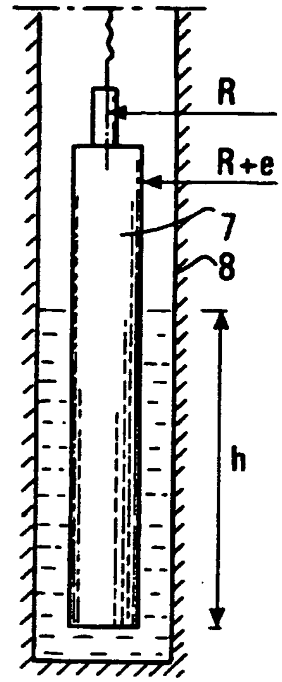


FIG.5

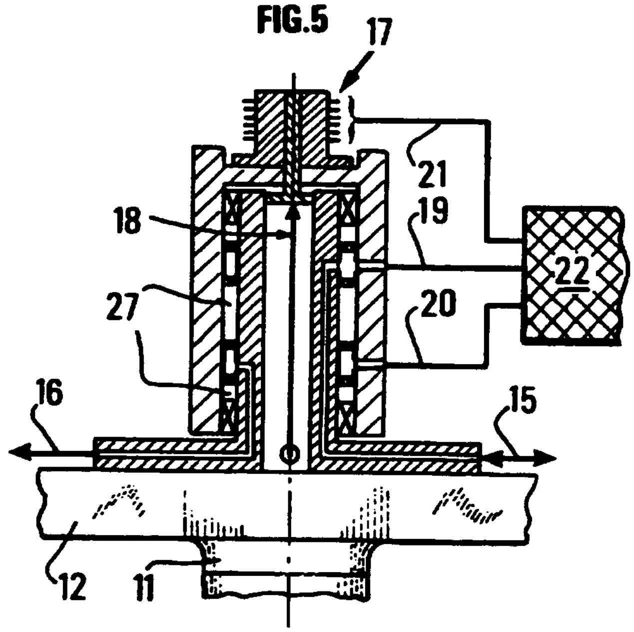


FIG.7

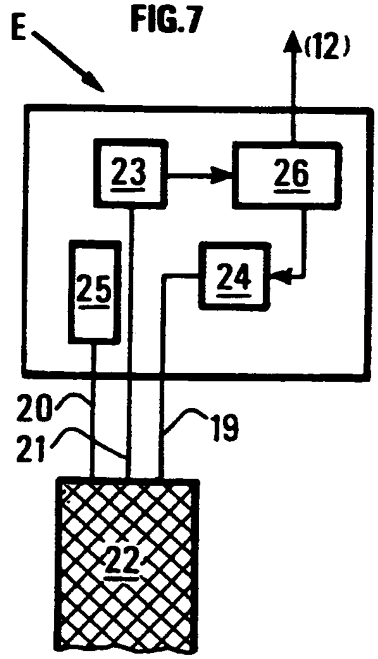


FIG.4

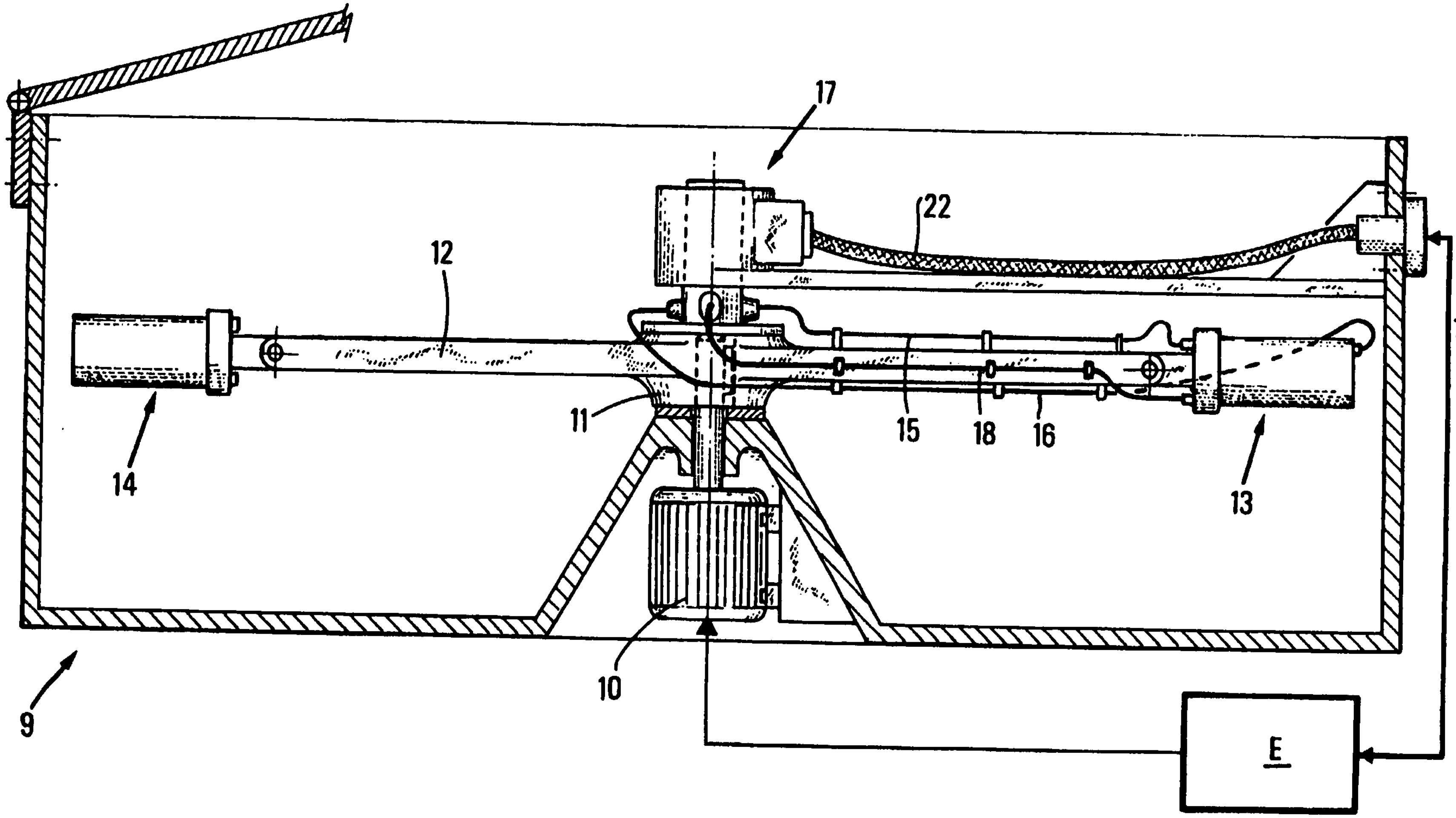
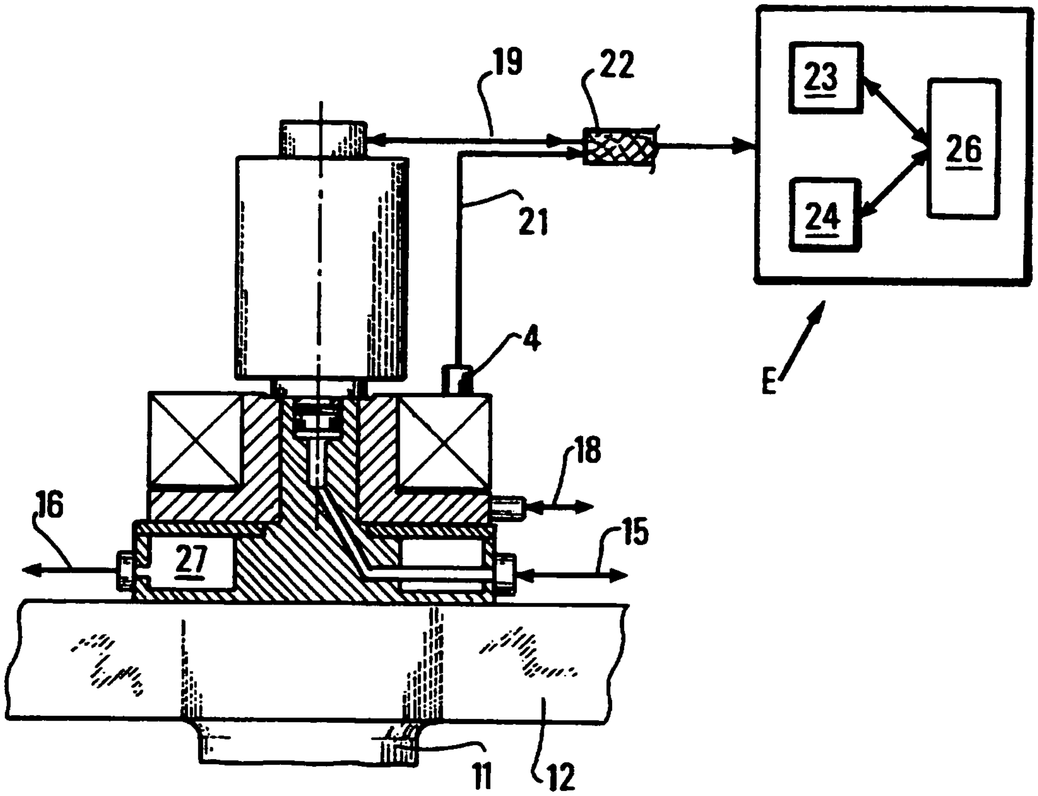


FIG.8



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DEVICE FOR MEASURING PHYSICAL CHARACTERISTICS OF A POROUS SAMPLE

The present invention relates to an improved device for measuring physical characteristics of a more or less porous sample. A tool of this type is particularly well suited to testing geological samples and determining various parameters such as the capillary pressure of the rocks in drainage or imbibition phases, their wettability indices, their relative permeability levels, resistivity indices, etc.

The device finds applications in the field of petroleum in particular, for testing rocks taken from formations containing or likely to contain petroleum effluents.

It is important to ascertain the wettability of the rocks by water and the oil that they might contain. To this end, it is necessary to drain the rocks, i.e. displace the fluids, in order to reduce water saturation, followed by a process of imbibition, this term being used to denote a displacement of the fluids as a means of increasing the water saturation (Sw) of the rock. The capillary pressure at a given point is defined as being the difference Pc at equilibrium between the pressure Po of the oil and that of the water Pw. This parameter is only meaningful if the two fluids are in continuous phase in the porous medium. In the case of a medium that is wettable by water, only positive values make sense. If the medium is of mixed wettability, on the other hand, the fluids may remain in continuous phase at both positive and negative capillary pressures (Pc).

In this type of application, the capillary pressure will therefore be measured by a complete cycle which will

include (Fig. 1):

a) positive primary draining of a sample initially 100% saturated with water (curve 1);

b) positive imbibition (curve 2);

c) negative imbibition (curve 3);

d) negative draining (curve 4; and

e) positive secondary draining (curve 5).

It is useful to know certain parameters and in particular the wettability of rocks, especially if enhanced recovery is to be used in a formation to drain the effluents contained in it by injecting a pressurised fluid, in which case tests will be carried out beforehand to determine which fluid (water or gas) is best suited to displacing the effluents.

The invention also finds applications in civil engineering for field hydrology as a means of evaluating the pollution level, for example, or in the building industry as a means of testing construction materials to decide on any waterproofing treatments that might be necessary, for example.

A method is known from patent FR-A-0.603.040 filed by the applicant which allows the physical characteristics of saturated rocks to be measured by subjecting them to centrifugation at progressive speeds and measuring the quantity of fluid displaced as a function of the rotation speed. The sample saturated with a liquid A, for example, is placed in an elongate recipient or container containing another fluid B of a different density. The container is secured to the end of a rotating arm and a centrifugal force is applied to it in order to study displacements of the fluids in the sample during at least two different phases. During a first drainage phase, the assembly is subjected to a

centrifugal force directed along the length of the container so as to exert an expulsion force on it which tends to cause the first fluid B to flow out. Some of fluid A simultaneously penetrates the interior of the sample. The two fluids are displaced within the sample until a position of equilibrium is reached or the force caused by the capillary pressure in the pores compensates for the centrifugal force applied.

It is known that the capillary pressure P_c at a distance R from the rotation shaft is expressed by the following equation when it is positive:

$$P_c(R) = \frac{1}{2}\Delta\rho\,\omega^2(R^2_{max}-R^2) \qquad P_c(R_{max})=0$$

where ω is the angular speed of rotation, R_{max} is the distance from the base of the sample bar S to the shaft and $\Delta\rho$ represents the differences in the respective densities of the two fluids.

For negative values, the capillary pressure P_c at the distance R from the rotating shaft is

$$P_c(R)=1/2\Delta\rho\,\omega^2(R^2_{min} - R^2) \qquad P_c(R_{min}) = 0$$

In the re-imbibition phase (curve 2), the speed is decreased in order to study the re-integration of the initial fluid therein. With this type of method, local saturations are calculated by an inversion programme using the total quantity of water expelled from the sample.

The capillary pressure in the sample can be derived by taking an accurate measurement of the initial quantity of fluid extracted as a function of the centrifugal force

applied and the variation in the mean saturation S_m of the sample by fluid as a function of the centrifugal force exerted, this latter being obtained by acoustic detection, for example.

With a sample saturated with a fluid (Fig. 1), saturation for a given radius r during the phase of drainage by centrifugation decreases (curve 1) as the rotation speed w is increased until it reaches a minimum value Si. During this drainage phase, the rotation speed is increased in successive steps to a level of 3500 rpm, for example. The variations in the saturation by fluid are measured during the deceleration phase. The occurrence of hysteresis is observed and a return along another curve of variation (curve 2) until a relative maximum Sm is reached during the re-imbibition phase of the porous material.

By preference, a system is used that will allow the drained fluid to be kept in contact with the sample bar so that when the deceleration phase starts, it can be re-imbibed in the bar correctly. In order to ensure that this contact is maintained, the systems stabilises the level of the interface between the two fluids at a minimum level at which it is flush with the base of the bar, i.e. as far away from the rotation shaft (R_{max}) as possible and does so at least during the entire deceleration phase.

The displacements within the sample are monitored, either by measuring the variation in flight time of the ultra-sounds through the sample or by measuring variations in its electrical resistance. The volume drained can be measured by optical means, using a vessel provided with a transparent port and observing the variation in level by means of stroboscopic lighting.

The fluids drained from the sample may be transferred into a chamber with a variable volume, for example, inside the vessel itself or in a second rotating container for example, by means of a pump mounted on one of the arms and driven by an electric motor. A system of this type can easily be set up and at a reasonable cost. However, it requires the use of pumping means mounted on the arm which means that it will be subjected to the centrifugal force. This is a disadvantage because it is difficult to find standard electric driving motors capable of withstanding the high accelerations needed to implement the method, typically some 3000 g. Special motors have to be used and these are expensive.

The improved device of the invention allows the physical characteristics of a solid, porous sample to be measured by setting up drainage and imbibition phases in succession in the presence of a first electrically-conductive fluid and a second fluid of a density lower than that of the first fluid. It incorporates an apparatus which can be moved in rotation and has at least one elongate recipient or container provided with an inner cavity for the sample, the container being secured to the end of an arm integral with a rotating shaft and linked to balancing means, and motor means to drive the arm in rotation and create a centrifugal force applied in the direction of elongation of the container.

The device is characterised in that it has a hydraulic system to force the displacement of the fluids and means for detecting the position of the interface connected externally to the detection means by a rotating connector, a capacitive sensor being provided in the container monitoring the direction of elongation thereof so as to monitor displacements of the interface between the two fluids in the container on a continuous basis.

The capacitive sensor has a metal rod, for example, coated with a fine layer of a dielectric material and the sensor is connected to a piece of apparatus for measuring the variation in the electrical capacitance of the sensor in contact with the fluids in the container due to the variation in the degree to which it is immersed in said conductive fluid.

In one embodiment, the device has a measuring and control system to control at least one transfer of fluid so as to keep the interface between the two fluids at a given level inside the container.

By preference, the measuring and control system is stationary and connected to the container by connector means incorporating a sealed, rotating electro-hydraulic connector and hydraulic passages as well as electrical connection means.

The system has, for example, a pump for the fluid of the lower density, a tank to collect at least some of the fluid expelled from the sample and a micro-computer programmed to acquire the signals issued by the apparatus and control fluid transfers in order to maintain the interface between the two fluids at a constant level during operation.

By preference, the micro-computer also has means for determining various physical parameters of the sample by taking account of the quantities of the two fluids displaced during operation.

In one embodiment, the device has a rotating electro-hydraulic connector with two sealed hydraulic channels, a first channel being connected to the

hydraulic system, and the tank is fixed and connected to the container by means of the second channel of said connector. Its contents are measured which will provide corroboration of the measured volume transferred by the pump.

In another embodiment, the device has a rotating electro-hydraulic connector with one sealed hydraulic channel connected to the hydraulic system and the tank for the first fluid is mounted on the rotating equipment and preferably positioned close to the rotating shaft.

In another embodiment, the device has a rotating electro-hydraulic connector with two sealed hydraulic channels providing communication between the hydraulic system and two symmetrically arranged containers driven in rotation by the motor means, said tank being integral with the mobile equipment and receiving the fluid from the two containers.

The measuring sensor used is highly accurate, detecting variations in the brine level to within 0.02 cc.

The device of the invention allows spontaneous imbibition of the sample drained during a prior centrifugation phase and thus provides accurate detection of the reverse displacement of the interface between the two fluids during the subsequent decrease phase until it is equal to zero. Its characteristics are very stable, particularly as regards rotation speed. The accuracy obtained by using the sensor to measure the level of the interface between the two liquids means that the saturation measurement on the sample is equally accurate, allowing the capillary pressure to be determined in the part of the bar between the interface and the face

closest to the rotating shaft. The sensor also takes up a reduced volume, which means that the container may be of a smaller size.

Other features and advantages of the invention will become clearer from the description of embodiments below, given by way of illustration and not restrictive in any respect, and with reference to the appended drawings, in which:

Fig. 1 shows various curves representing variations in the saturation of a sample during a drainage and imbibition cycle;

Fig. 2 is a schematic illustration of the layout inside a container with its sensor for monitoring the level of the interface between the two fluids;

Fig. 3 is an example of the variation in percentage (IL) of the signal issue by the measuring assembly as a function of the height H (and the volume IV) of brine in the container;

Fig. 4 is a schematic illustration of the layout of a centrifuge and pumping means external to the rotating assembly;

Fig. 5 is a schematic illustration in section of the layout of an electro-hydraulic connector used to link a container to an external control and acquisition unit;
Fig 6 illustrates the calculation of the sensor capacitance as a function of the variation in the level of the conductive liquid discharged from the sample during operation;

Figure 7 is an operating diagram of the external assembly

E for controlling and acquiring the measurement signals; and

Fig. 8 is another embodiment using a rotating electro-hydraulic connector with a single hydraulic circuit.

The device of the invention has (Fig. 2) has an elongate recipient or container 2 for a sample 1 of porous rock initially saturated with an electrically conductive fluid A such as brine, for example. The container 2 is initially filled with another, electrically insulating fluid B such as oil. At its opposing ends, the sample 1 in its container 2 (Fig. 2) is supported against two perforated disks 3 made from a porous ceramic, for example. The container 2 also has a tubular, lateral chamber 4 parallel with the direction of elongation of the sample and the container for the level-detector sensor 5 of the capacitive type.

This sensor 5 has (Fig. 6) a first electrode 6 which is a metal rod coated with a fine layer 7 of a dielectric material such as Teflon® or a glass ceramic, for example, and a second, bare metal electrode 8, the potential of which is used as a reference.

The capacitance between the electrodes is expressed as follows:

$$c = 2\Pi\varepsilon_0\varepsilon_r\left[\ln\frac{R+e}{R}\right]^{-1} h \approx 2\Pi\varepsilon_0\varepsilon_r\frac{R}{e}h,$$ where

R is the radius of the rod 6,

e is the thickness of the sheath or coating 7 covering the rod,

ε_r is the relative dielectric constant of the material of the sheath,

γ_0 is the dielectric constant of the vacuum, which is 8.859 10^{-12} A.s/V.m

Any variation in the level of the conductor fluid in the container is translated by a variation in the capacitance of the sensor. By using an electrode with a 3 mm radius and an insulating coating of 0.05 mm, a range of variation in capacitance of the sensor can be obtained between 10 and 1000 pF, for example. Before use, the sensor is calibrated by indicating to the associated measuring unit the minimum level (0%) and the maximum level (100%) between which the level of brine may be displaced within the container during operation. This allows the response curve of the sensor to be established, as illustrated in Fig. 3.

The centrifuge has a tank 9, an electric motor 10 whose shaft is driven in rotation by a hub 11. Two identical arms 12 are mounted opposite one another on the hub 11. Two recipients or containers 13, 14 are pivotally mounted on the ends of the two arms 12 so that they will align spontaneously with the direction of the centrifugal force applied and these balance one another in rotation. The sample to be measured is placed in the container 13.

Two passages 15, 16 fixed along one of the arms 9 link the container 13 to a rotating electro-hydraulic connector 17 borne by the hub 11. The first passage 15 is used to inject oil into the container 13. The second, 16, is used to collect the brine which is drained out from the sample by the force of rotation. A cable 18 also connects the measuring sensor 6-8 to the rotating connector 17. The connector 17 provides communication (Fig. 5) for the two rotating passages 15, 16 with two hydraulic ducts 19, 20 and the conductors of the cable 18 are electrically connected to another cable 21. These two

passages and this cable form a linking element 22 between the container 13 and an external unit E.

This unit has a measuring device 23 of a known type connected to the cable 21 and issues a signal proportional to the height of the sheathed electrode immersed in the conductor liquid and, since the section of the lateral chamber is known, the corresponding variation in volume, an oil injection pump 24 connected to the passage 19, a tank 25 for the brine connected to the passage 20 and a controlling micro-computer 26 fitted with an interface card and programmed to control the pump 24 and the motor 10 of the centrifuge and to acquire the measurements issued by the measuring device.

To produce a very high degree of accuracy in the fluid transfers, a pump 24 and a rotating connector 17 were selected with special seals to ensure minimum leakage within a broad operating temperature range at pressures in the order of 0.5 Mpa.

A sample saturated with brine, for example, is placed in the container and the connecting circuit 18, 20 to the tank 25 is filled with brine. The centrifuge is started at a minimum speed of 200 rpm. Oil is then pumped into the container via the circuit 15, 19 until the 0% minimum level is reached corresponding to the position of the external face of the sample for which the sensor 5 was calibrated. The level of brine drained out from the sample rises slowly as the rotation speed of the centrifuge is increased to approximately 1000 rpm, for example. It is measured by the level detector 23 (Fig. 7) and transmitted to the computer 26 which controls the injection of pressurised oil so as to restore the initial level of 0%. The brine is therefore discharged through the circuit 16, 20 into the tank 25 where its level

rises.

When the rotation speed decreases (imbibition phase), the oil pump 24 is activated in order to top up with just enough oil to maintain the level of the sensor at its lowest reference point of 0%. At the same time, the brine in the tank 25 is returned so as to be re-imbibed in the sample.

The following data are acquired during the drainage-imbibition cycle:

the volume of oil pumped into the container or out of it, which gives the mean saturation of the sample;

the rotation speed which is related to the capillary pressure;

the position of the interface level inside the container.

The mean saturation of the sample can also be obtained from the level of brine 25 in the tank and the position of the pump. Optionally, the variation in the brine level 25 inside the tank can also be measured, which should confirm the recorded volume of oil pumped into the container.

The micro-computer may also be programmed to calculate petro-physical parameters directly from the measurements taken during the drainage and imbibition cycles.

In the embodiment illustrated in Fig. 8, a rotating electro-hydraulic connector with only one hydraulic channel is used. The pump 24 in the stationary, external unit E delivers the less dense fluid under pressure via

passages 15 and 19 connected to one another by the rotating connector 17. Here, the passage 16 links the end of the container farthest from the rotation shaft to an auxiliary tank 27 which is also driven in rotation. This tank 27 is annular, for example, and is fixed to the rotating hub 11.

In one embodiment, the tank 27 may also be an auxiliary cavity of the same container 13, as already described in patent FR-A-2.603.040, mentioned above.

The fluid produced (the denser or the less dense, as is the case) is kept in contact with the sample during centrifugation. Consequently, the expelled fluid is able to re-integrate the sample when the imposed pressure is decreased. Depending on the type of experiment being conducted, the interface between the two fluids is kept in contact with the first end of the sample (the farthest from the rotation shaft) or its opposite end (the closest to the rotation shaft).

When the capillary pressure values are negative, the interface between the two fluids is maintained at a stable level, in the vicinity of the first end of the sample and the position is controlled by means of the measurement signal issued by the level analyser 23 and the rotation speed is increased in stages to produce drainage and then decreased in stages to initiate spontaneous imbibition.

When the capillary pressure values are positive, on the other hand, the interface between the two fluids is kept close to the second end of the sample (that closest to the rotation shaft) and the rotation speed is accelerated in stages to force imbibition.

As above, the sample S is saturated with brine, for example. The centrifuge is maintained at a minimum rotation speed (200 rpm for example). Oil is then injected into the container by means of the pump 24 until it reaches the reference level in the vicinity of the first end, which can be controlled by the readings from the capacitive sensor 5. As the speed is increased, the brine is expelled from the sample. To maintain the interface stable, pressurised oil is injected via the passage 15. The excess brine flows through the passage 16 into the tank.

At decreasing rotation speeds on the other hand, the pump, controlled by the capacitive sensor, draws off oil to maintain the reference level selected for the interface and the brine that has collected in the tank R is re-integrated into the container 13.

Once the rotation speed has returned to the minimum level, the interface level is positioned in the vicinity of the second end of the sample (the closest to the shaft) and the process may proceed with a forced imbibition phase, taking care, as above, to ensure that this reference level is maintained, without the need to remove the sample from its container.

The data acquired during the different phases are the volume of fluid pumped into or out of the container which will give the mean saturation of the sample, the rotation speed of the centrifuge which will give the capillary pressure and the position of the interface level.

If the device has an electro-hydraulic connector with two hydraulic channels as illustrated in Fig. 5, tests can be conducted simultaneously on samples placed

in the two symmetrical containers 13, 14 and the brine drained simultaneously from the two containers will be collected in the same tank 27.

The stability of the signal issued by the device 23 was verified for a same brine level in the container in relation to three causes of instability: temperature variation, induced noise generated by the rotating contacts, other effects due to rotation. The test results show that the measuring errors of the sensor are limited to 1% of the full scale, which represents a measurement to less than the nearest mm of the height of brine in the container.

An embodiment is described (Fig. 8) in which a rotating electro-hydraulic connector with one hydraulic channel is used in which the rotating part (rotor) is at the centre of a stationary ring. However, it would not be a departure from the scope of the invention if a connector of another known type were used in which the external ring were secured to the hub 11 and rotor and the central piece were fixed.

CLAIMS

1. An improved device to a device for taking measurements of physical characteristics on a solid porous sample by operating drainage and imbibition phases in succession in the presence a first, electrically-conductive fluid and a second fluid of a density lower than that of the first fluid, provided with a piece of apparatus that can be moved in rotation having at least one elongate container with an inner cavity for the sample, the container being fixed to the end of an arm integral with a rotating shaft and linked to balancing means, motor means to drive the arm in rotation and create a centrifugal force along the direction of elongation of the container, characterised in that it has a system for forcing the displacement of at least the fluid having the lower density and means for detecting the interface position externally connected to detection means by a rotating connector, having a capacitive sensor arranged in the container along the direction of elongation thereof to monitor continuously the displacements of the interface between the two fluids inside the container.

2. A device as claimed in claim 1, characterised in that the capacitive sensor comprises a metal rod, for example, coated with a fine layer of a dielectric material, the sensor being linked to a device for measuring the variation in the electrical capacitance of the sensor in contact with the fluids inside the container due to the varying degree of immersion thereof in said conductor fluid.

3. A device as claimed in one of the preceding claims, characterised in that it has a measuring and control system (E) for controlling at least one transfer

of fluid in order to maintain the interface between the two fluids at a given level inside the container.

4. A device as claimed in the preceding claim, characterised in that the measuring and control system (E) is stationary and connected to the container by connection means incorporating a sealed, rotating electro-hydraulic connector as well as hydraulic passages and electric connecting means, the system (E) comprising a pump for the lower-density fluid, a tank to collect at least some of the fluid expelled from the sample and a micro-computer programmed to acquire the signals issued by the device and control the fluid transfer in order to maintain the interface between the two fluids at a constant level during operation.

5. A device as claimed in one of the preceding claims, characterised in that it has a rotating electro-hydraulic connector with two sealed hydraulic channels, a first channel being connected to the hydraulic system, the tank being stationary and connected to the container via the second channel of said connector.

6. A device as claimed in one of claims 1 to 4, characterised in that it has a rotating electro-hydraulic connector with at least one sealed hydraulic channel connected to the hydraulic system, said tank being integral with the moving apparatus.

7. A device as claimed in claim 6, characterised in that it has a rotating electro-hydraulic connector with two sealed hydraulic channels providing communication between the hydraulic system and two containers arranged symmetrically and driven in rotation by motor means said tank being integral with the moving apparatus and receiving the fluid from the two containers).

8) A device as claimed in one of claims 3 to 7, characterised in that the measuring and control system (E) has means for determining different physical parameters of the sample taking account of the quantities of the two fluids displaced during operation.

9) A device for taking measurements of the physical characteristics on a solid porous sample substantially as hereinbefore described with reference to figures 2 to 7 of the drawings.

10) A device for taking measurements of physical characteristics on a solid porous sample substantially as hereinbefore described with reference to figure 8 of the drawings.

The Patent Office

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Application No:	GB 9810801.2	**Examiner:**	Michael Walker
Claims searched:	ALL	**Date of search:**	9 September 1998

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.P): G1S (SGX, SIA)

Int Cl (Ed.6): G01N 15/00, 15/08; 33/24

Other: ON-LINE : WPI

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
Y	US 5463894 (FLEURY) whole document	1,2,4,8
Y	US 5035583 (VAUGHT) see abstract	1,2,4,8
Y	WPI Accession No.93-161304/199320 & FR 2681424 A1 (CHOISNET et al.) 19.03.93 (see abstract)	2
Y	WPI Accession No.88-339077/198848 & DE 3741569 C (LOTHMANN) 01.12.88 (see abstract)	2

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.